PATENT

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Dated: September 27, 2007 BY: Rodney D. DeKruif

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: Emrick et al.	)	
Serial No: 10/643,015	)	Attorney Docket No. 7163
Filed: August 18, 2003	)	
For: PYRIDINE AND RELATED LIGAND COMPOUNDS, FUNCTIONALIZED NANOPARTICULATE COMPOSITES AND METHODS OF PREPARATION	)	

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

RULE 131 DECLARATION OF HABIB SKAFF

1. I, Habib Skaff, am a co-inventor with regard to the invention (the "Invention") disclosed and claimed in the above-entitled application (the "Application"). I make this declaration in support of the Application and, in particular, to antedate a reference cited against the Application.

2. The Invention claimed in the Application was completed before the effective date of application serial number 10/219,440 (*i.e.*, the Dubertret

reference). More specifically, the Invention was conceived and with due diligence reduced to practice, in this country--the United States of America, prior to the effective date of the Dubertret reference.

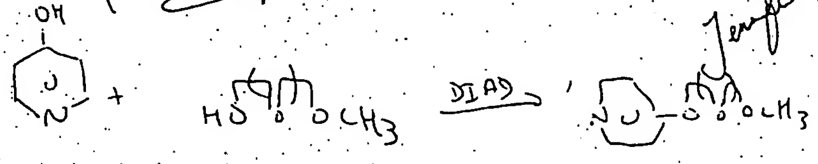
3. This Declaration, and prior invention, is supported by copies of pertinent pages from my laboratory research notebook, entries to which I contemporaneously signed and dated and were witnessed by co-inventor, Todd S. Emrick. Date redacted copies of the aforementioned notebook pages are provided collectively as Exhibit A and incorporated herein by reference. These documents establish that the Invention was made at least as early as June 1, 2002, which is a date earlier than the effective date of the Dubertret reference. Without limitation, facts demonstrating prior invention of a composite of independent claim 1 include the experimental data I entered on page 37 of Exhibit A. Facts demonstrating prior invention of a system of independent claim 14 include the experimental data I entered on page 37 of a Exhibit A. Facts demonstrating prior invention of a method of independent claim 20 include the experimental data I entered on page 38 of Exhibit A.

I hereby declare that: All statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; that those statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code; and that willful false

statements may jeopardize the validity of the Application or any patent issuing thereon.

Date 9/24/07

Habib Skaff



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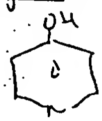
Reagents

95 ①  2g, 0.022 mol

③

750 ② m-Pg 750 14.25g, 0.019 mol

262 ③ Ph_3P 6.28g, 0.024 mol

202 ④ DIAD 4.84g, 0.024 mol (4.72 mL)

⑤ THF (dry) ~~300 mL~~ 250 mL

Procedure

① Ph_3P + THF loaded into 2-neck flask & stirred under N_2 @ r.t.

② DIAD added via syringe & stirred for ½ hr.

③ phenol & alcohol added & stirred

④ reacted over night

⑤ rotavaped off THF

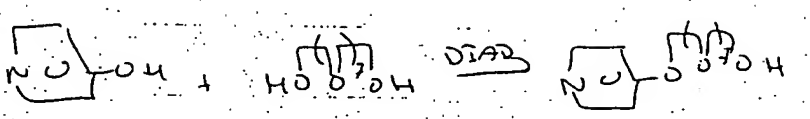
⑥ added H_2O & ether ⇒ washed w/ ether,

⑦ extracted product out w/ CH_2Cl_2 out of Aq phase ⇒ $MgSO_4$, Rotovap

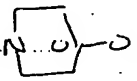
→ NMR show some (pyridinol) ⇒ tried redissolving in basic (.5M NaOH) solution & precipitating into CH_2Cl_2 (cold) → didn't work

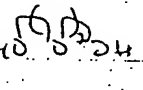
→ ran column eluting w/ CH_2Cl_2 : [illegible] : MeOH (7:3:0), (7:2:1)

Exhibit A



Reagents

95 ①  & 2g , 0.011mol

400 ρ=1.03 ②  22g , 0.055mol

202 ③ DIAD 2.63g , 2.55mL 0.013mol

262 ④ Ph_3P 3.41g , 0.013

⑤ THF(dry) 300mL

Procedure

① Ph_3P & THF loaded into 3-neck 500mL round bottom ; stirred @ rt under N_2

② DIAD added via syring ; stirred for ½hr.

③ phenol ~~& diol~~ added ; stirred

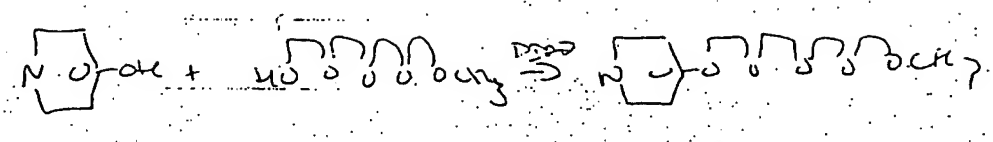
→ reacted overnight

- rotovaped off all THF → oil

- extracted w/ H_2O → then aqueous washed w/ CH_2Cl_2 → too difficult to purify by column

→ rotovaped off CH_2Cl_2 → dissolved in H_2O, washed w/ ether, then Toluene → doesn't work well

- try acidifying aqueous to make pyridine salt which will not be soluble in



Reagents

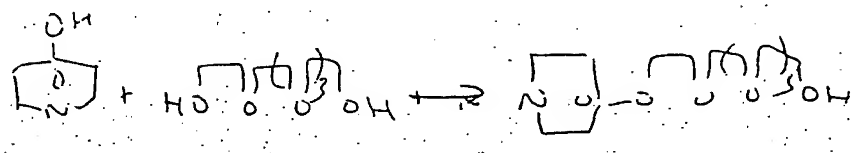
95	①	5 g,	0.055 g mol
128	② m-T-g	5.632 g,	0.044 mol
262	③ Ph_3P	13.1 g	0.05 mol
202	④ DIAD	10.1 g,	0.05 mol, 9.85 mL
	⑤ THF (dry)	~~75 mL~~	400 mL

Procedure

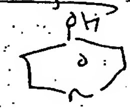
① ~~5 g~~ Ph_3P & THF loaded into 2-neck flask & stirred under N_2 @ r.t.

② DIA added via syring & stirred for ½ hr.

③ phenol & alcohol added & stirred overnight



Reagents

95 ①  4g, 0.042mol

300 ② Heg 31.58g, 0.105mol

262 ③ Ph_3P 13.1g ~~0.045~~ 0.05mol

(=1.05) 202 ④ DIAD 10.1g, 0.05mol, ~~10.35mL~~ 9.85mL

⑤ THF 500mL

Procedure

① phenol, Ph_3P, DIAD, & THF loaded in 2-neck & stirred @ r.t. under N_2 for ½ hr.

② diol added ⇒ stirred overnight

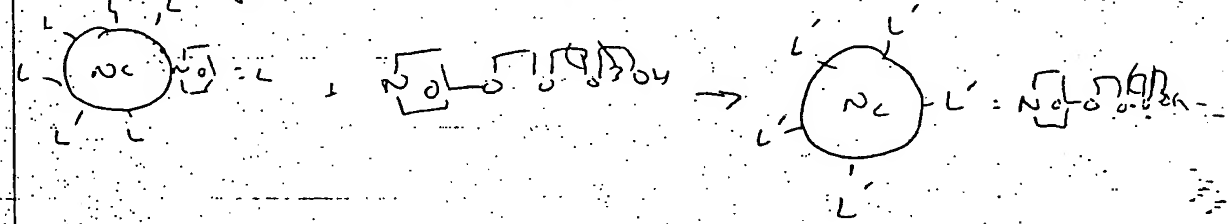
rotavaped off THF
⇒ first w/ $CHCl_3$ ② $CHCl_3$: (80:20) ③ ~~$CHCl_3$~~ $CHCl_3$: : MeOH (75:20:5)
⇒ ran column eluting w/ ④ $CHCl_3$: : MeOH (7:2:1)

⇒ tried distilling off unreacted diol @ ~~180~~ 224 °C @ @ 600 mtorr ⇒ didn't work well

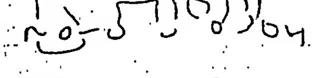
⇒ ran column in $CHCl_3$: : MeOH (78:20:2), (75:20:5), (80:20:10)

[signatures]

Exchange to teg-monopyridinyl ether



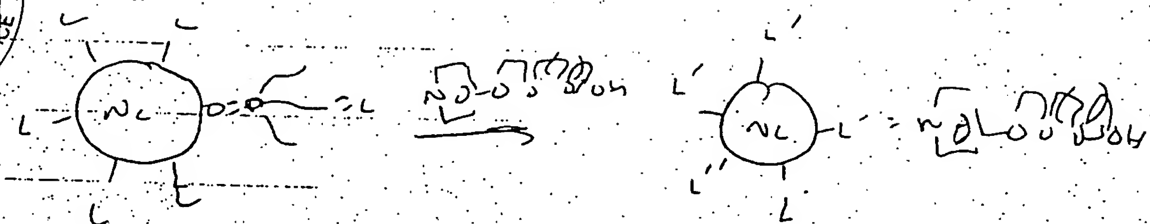
Reagent

1) pyridine NC ~40mg
2)  600mg
3) THF (dry) 3mL
4) DIW 6mL

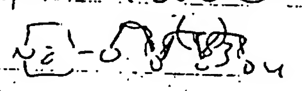
Procedure

A) ① 20mg NC dispersed in solution of 300mg new ligand in THF → immediately went it into solution
② dried under N_2 flow and added 3mL DIW → most went into solution → centrifuged

B) ③ 20mg NC dispersed in solution of 300mg new ligand in 3mL DIW → NC went into solution → centrifuged



Reagents

① TOPO covered NC ~15 mg
②  320 mg
③ THF (dry) 3 mL

Procedure

① NC made as ~~usually~~ usual & washed w/ MeOH 3 times
② dried over N_2 flow
③ redissolved in new ligand in THF and allowed to stand over head of N_2 overnight
④ distilled off ½ THF → precipitated w/ hexane → all NC precipitated
⑤ washed w/ hexanes → centrifuged → redissolved in ~~DP~~ H_2O

[signature] [signature]

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